# THE RESTEK ADVANTAGE

Turning Visions into Reality™

2005 vol. 4


## Enhanced Electronic Leak Detector

### A Leak-Free System Stabilizes Baselines and Lengthens Column Life

new for 2005

By Donna Lidgett, GC Accessories Product Marketing Manager

- Reliable thermal conductivity leak detector—every analyst should have one.
- Compact, portable, ergonomic design, easy to hold and operate.
- Sensitive—detects helium or hydrogen at $1x10^{-4}$ cc/sec*.
- Fast results—responds to leaks in less than 2 seconds, zeros with the touch of a button.
- Built-in rechargeable battery; charging adaptor included.


In continuing our efforts to provide the best available columns, tools, and accessories, we have enhanced our popular leak detector. New features include internal battery charge capability, a low-battery indicator, a battery charge indicator light, yellow lights to signal a nitrogen leak, a repositioned on/off switch, to eliminate accidentally powering on the unit, and a new probe tip design that prevents debris from entering the unit, and allows the tip to be removed for easy cleaning. The new leak detector retains the microchip technology that enables high sensitivity in a compact unit, instantaneous zeroing with the touch of a button, and the ergonomic design that puts all controls at your fingertips, for maximum ease of use.

The new Restek Electronic Leak Detector is the affordable solution for detecting helium, hydrogen, or nitrogen leaks in your GC system. Leaks can cause detector noise and baseline instability, waste carrier gas, and shorten column lifetimes. The leak detector responds in less than 2 seconds to leaks of gases with thermal conductivities different from air, indicating leaks with both an audible alarm and an LED readout. The leak detector detects minute gas leaks that can go undetected by liquid leak detectors. And, remember—you should never use liquid leak detectors on a capillary system, because liquid drawn into the system through the leaks will contaminate the system.

| Description | qty. | cat.# |
|---|---|---|
| Leak Detector with 110Volt Battery Charger | ea. | 22451 |
| Leak Detector with 220Volt European Battery Charger | ea. | 22451-EUR |
| Leak Detector with 220Volt UK Battery Charger | ea. | 22451-UK |

Caution: The Restek Electronic Leak Detector is NOT designed for determining leaks of combustible gases. Use a combustible gas detector for determining combustible gas leaks in possibly hazardous conditions.

*Sensitivity measured using helium.

## in this issue



## Looking Ahead

**2006 Restek Catalog**

We've added many new GC columns, HPLC columns, tools, supplies, and reference mixes to help make life in your laboratory a little less complicated. Look for your copy in your mail in January. If you currently don't receive Restek literature, and would like to have a catalog, simply go to our website and sign up for one **www.restek.com/catalog**.

**Pittcon® 2006**

Time to make reservations, if you haven't done so already. Pittcon® 2006 will be held in Orlando, Florida, March 12-17. We look forward to talking with you, and showing you our latest innovations in chromatography. You can read about some of them in our next issue of the Advantage.

**Special Note:**

In September and October, Restek customers and Restek employee-owners, with matching donations from Restek, contributed $9845.10 to hurricane relief. Please remember the survivors of 2005's worldwide catastrophes this holiday season.

# Superior Separations of Unsaturated Compounds by HPLC

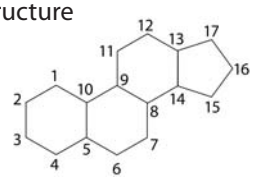
## Separating Steroids by π-π Interactions Using the New Allure™ Biphenyl Column

By Rick Lake, Pharmaceutical Applications Chemist, Randy Romesberg, HPLC Applications Chemist, and Mike Wittrig, R&D Chemist

- Greater retention and specificity for compounds with small differences in double bonding.
- Better resolution, efficiency, and specificity for steroids, compared to C18 phases.
- Excellent choice for stability-indicating methods.

Steroids owe their broad range of medicinal properties largely to the diversity in their chemical structures. The basic structure consists of a phenanthrene ring linked to a cyclopentane ring (Figure 1), but various levels of unsaturation (double and triple bonds) and differing ring substituents (functional groups) create great diversity. Because the diversity in steroid composition consists of variations from one structure, we chose these compounds to illustrate the superiority of the Allure™ Biphenyl stationary phase for analyzing unsaturated compounds.

**Figure 1** Basic Steroid Structure




Steroids are hydrophobic molecules that typically are analyzed using a reversed phase column, such as a C18 column. The hydrophobic surface of this phase interacts with the hydrophobic portions of analyte molecules. This provides adequate separation for steroids that have differing hydrophobicity and differing functional groups. As Figures 2-4 show, however, the hydrocarbon ring system also presents structural variations. A separation mechanism based primarily on hydrophobic characteristics has limited effectiveness for resolving unsaturated compounds that differ only in the location of double bonds in a carbon ring.

In contrast, the new Allure™ Biphenyl stationary phase offers a unique separation mechanism that is more selective for separating compounds with slight differences in saturation: pi-pi (π-π) interactions. These interactions can occur when the ring moieties of the steroids and the biphenyl phase overlap.

We conducted three separate analyses, using simple isocratic conditions. Hydrocortisone, cortisone, and prednisone, which differ in C1-C2 double bonding and exhibit slight differences among position 17 and 11 functional groups, are almost completely resolved by an Allure™ Biphenyl column (Figure 2). The C18 stationary phase is unable to resolve hydrocortisone and prednisone.

**Figure 2** Greater retention and resolution of corticosteroids, using an Allure™ Biphenyl column.

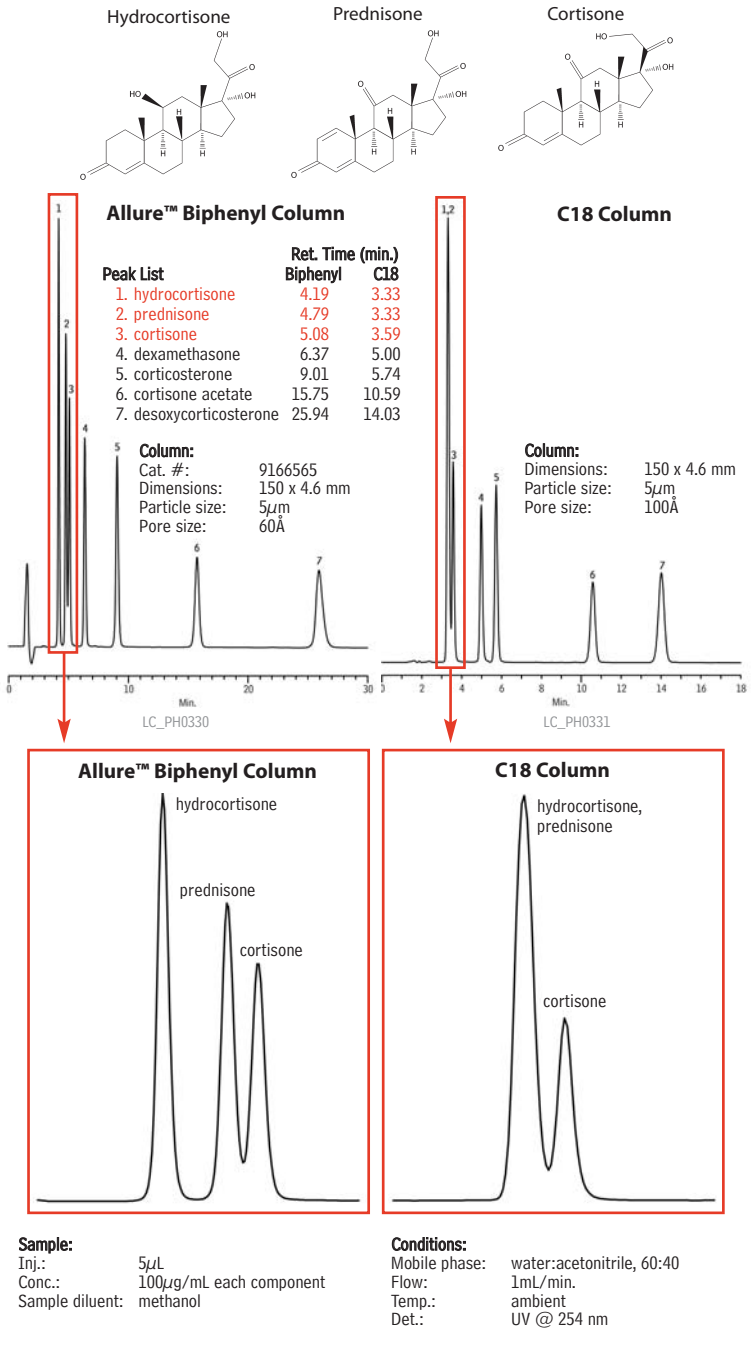
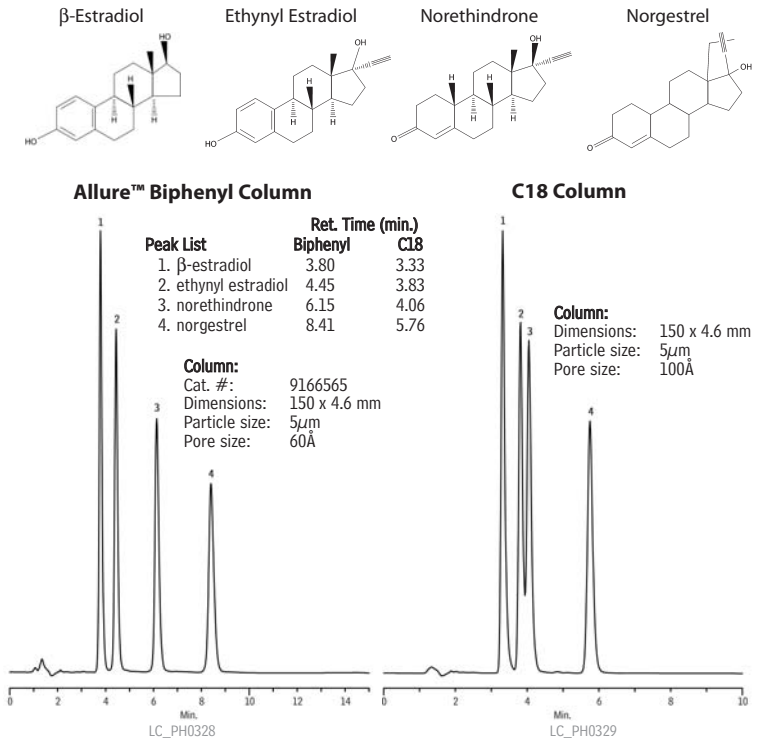


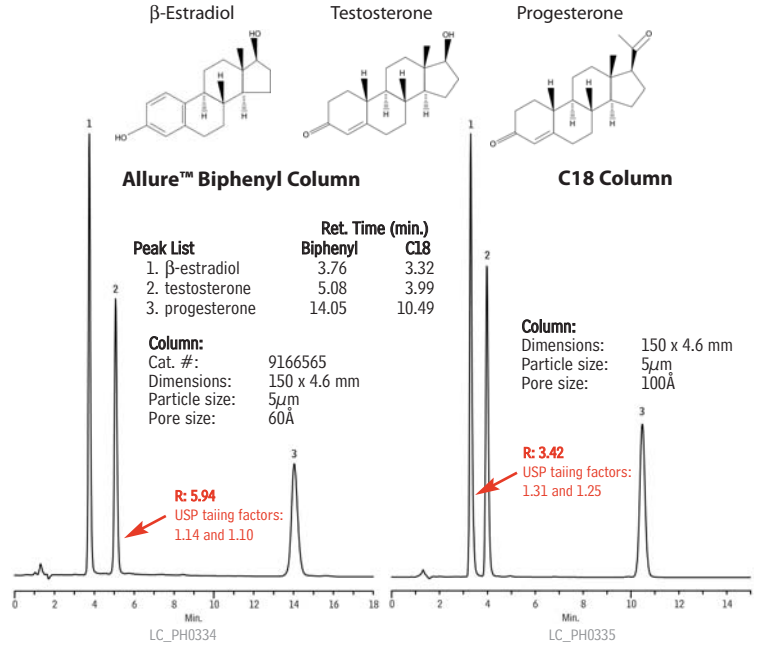
**Figure 3** Baseline resolution of contraceptive steroids on an Allure™ Biphenyl column.




**Sample:**
Inj.: 5µL
Conc.: 100µg/mL each component
Sample diluent: methanol

**Conditions:**
Mobile phase: water:acetonitrile, 40:60
Flow: 1mL/min.
Temp.: ambient
Det.: UV @ 220 nm

**Figure 4** An Allure™ Biphenyl column provides greater resolution and peak symmetry for endogenous steroid hormones.




**Sample:**
Inj.: 5µL
Conc.: 100µg/mL each component
Sample diluent: methanol

**Conditions:**
Mobile phase: water:acetonitrile, 40:60
Flow: 1mL/min.
Temp.: ambient
Det.: UV @ 220 nm

The minor differences in ring structure are sufficient to enable the Allure™ Biphenyl phase, but not the C18 phase, to elute these steroids selectively.

Contraceptive hormones also illustrate the Allure™ Biphenyl phase's superior retention and selectivity for steroids (Figure 3). As expected, the C18 phase resolves β-estradiol and ethynyl estradiol, which have differing functional groups, but it cannot resolve ethynyl estradiol and norethindrone, which have differing ring structures.

To verify the selectivity of the Allure™ Biphenyl phase, and to investigate possible enhanced system suitability criteria, we also analyzed endogenous hormones. β-estradiol and testosterone are structurally very similar, differing primarily in ring structure (Figure 4). By comparing resolution of these two compounds, we can make a correlation between hydrocarbon ring variation and resolution. The C18 column produced a resolution of 3.42, with USP tailing factors of 1.31 and 1.25, respectively; the Allure™ Biphenyl column provided a resolution of 5.94 — a 43% increase — and superior tailing factors of 1.14 and 1.10.

In all these analyses, the Allure™ Biphenyl column provided superior retention factors, relative to the C18 column. By increasing retention, we increase selectivity — the most effective way to improve resolution among analytes. When selecting a column for steroids, or for any other analysis, the stationary phase that provides greater retention ultimately will allow more control in choosing other method parameters and, thus, more control over the analysis.

Overall, these analyses demonstrate that π-π interactions are an excellent mechanism for resolving compounds with saturation differences in their hydrocarbon structures. The Allure™ Biphenyl stationary phase offers excellent retention, selectivity, and efficiency for unsaturated compounds with or without unsaturated functional groups. The greater selectivity and efficiency exhibited by the Allure™ Biphenyl phase, relative to a C18 phase, make it well suited for developing stability-indicating methods. Greater selectivity can mean better resolution between an analyte and its degradation products; greater efficiency will provide the capability to institute more accurate system suitability parameters.

**Allure™ Biphenyl Column**

| 5µm Column, 4.6mm | cat. # |
|---|---|
| 150mm | 9166565 |

## for **more** info

For more information, and a complete list of Allure™ Biphenyl columns, request lit. cat.# 580015.

# Sub-ppb GC/MS Analysis of MTBE and TBA

## Using an Rtx®-VMS Column and an OI 4660 Purge & Trap System

By Jason Thomas, Environmental Innovations Chemist

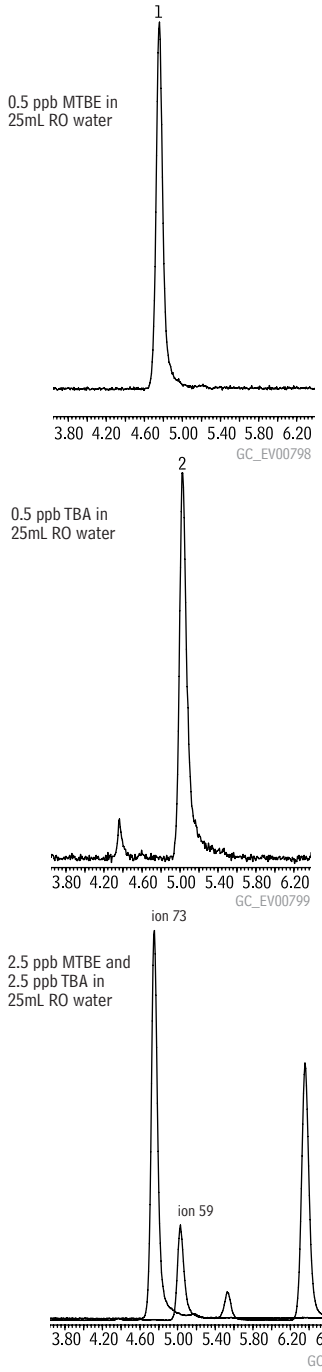
- Optimized analysis for common oxygenates.
- Excellent reproducibility to 0.5ppb.
- Unique Rtx®-VMS phase fully resolves MTBE & TBA.

In an attempt to reduce automotive emissions in the US, the Clean Air Act Amendments of 1990 require that gasoline formulations contain at least 2% oxygen. Initially, addition of 15% methyl *tert*-butyl ether, MTBE, to gasoline was a common way of meeting this requirement. This became a concern, however, as MTBE has a relatively high solubility in water and, therefore, can easily contaminate groundwater sources. Subsequently, *tert*-butyl alcohol, TBA, has been included as a target compound on the oxygenates list, as it is both a contaminant in MTBE and a breakdown product of MTBE. Because of its appreciable affinity for water (greater than MTBE, and harder to detect than MTBE using purge and trap), and its potential health hazards, it also must be monitored.

To analyze for MTBE and TBA simultaneously, the two compounds must be resolved chromatographically, because the minor ion 59 of MTBE interferes with the quantification of TBA, whose primary ion is 59. The two tend to co-elute on many "624-type" columns (6% cyanopropylphenyl / 94% dimethyl polysiloxane stationary phases), making this separation a challenge.

The unique Rtx®-VMS phase provides the solution to this problem, resolving MTBE and TBA (Figure 1) and contributing to reliable quantification of both compounds. Using an OI 4660 Eclipse purge and trap system and an Rtx®-VMS column (cat.# 19915), optimizing purge and trap conditions (65°C sample temperature) to increase the concentrations of the low molecular weight oxygenates on the trap and allow the best responses at low levels, and operating the mass spectrometer in the SIM mode, we detected both compounds accurately at concentrations as low as 0.5ppb in 25mL water (Figure 1). Over a concentration range of 0.5ppb to 5ppb, we achieved relative standard deviations of 5.2% for MTBE and 8.7% for TBA. Based on these results, we highly recommend an Rtx®-VMS column for low level purge and trap analysis of MTBE and TBA, in conjunction with the standard Method 8260 analysis.

**Figure 1** An Rtx®-VMS column resolves MTBE and TBA, for reliable quantification of both compounds.




**Column:** Rtx®-VMS, 30m, 0.25mm ID, 1.4µm (cat.# 19915)

**Purge and Trap**

| | |
|---|---|
| Model: | OI 4660 Eclipse Purge and Trap |
| Trap: | #10 (Tenax®/silica gel/carbon molecular sieve) |
| Sample temp.: | 65°C |
| Purge: | 11 min. @ 40mL/min. |
| Desorb preheat: | 185°C |
| Desorb: | 0.5 min. @ 190°C |
| Desorb flow rate: | 35.0mL/min. |
| Bake: | 6 min. @ 210°C |
| Interface: | split injector |
| Transfer line: | 2m, 0.25mm ID Siltek® treated tubing (cat.# 22502), temp.: 150°C |

**Injection**

| | |
|---|---|
| Split ratio: | 35:1 |
| Inlet liner: | 1mm ID Siltek® treated splitless (cat.# 20972-214.1) |
| Inj. temp.: | 200°C |

**Oven**

| | |
|---|---|
| Carrier gas: | helium, constant pressure |
| Linear velocity: | 36cm/sec. @ 35°C |
| Oven temp.: | 35°C (hold 7 min.) to 70°C @ 4°C/min. (hold 3 min.) to 220°C @ 20°C/min. (hold 3 min.) |

**Detector**

| | |
|---|---|
| Det.: | MS |
| Transfer line temp.: | 150°C |
| Scan range: | 73, 59, 57, & 41 amu |
| Ionization: | EI |
| Mode: | SIM |

1. methyl-*tert* butyl ether (MTBE)
2. *tert*-butyl alcohol (TBA)

### for more info

For reference mixes see **page 13** and our website.

For a purge an trap analysis of petroleum oxygenates, including ethanol, visit our website (search: GC_EV00401).

### Rtx®-VMS Column (fused silica)

(proprietary Crossbond® phase)

| ID | df (µm) | temp. limits | length | cat. # |
|---|---|---|---|---|
| 0.25mm | 1.40 | -40 to 240/260°C | 30-Meter | 19915 |

# Analyzing Oxygenates in Gasolines

## Using a Deactivated Sample Pathway and ASTM Method D-4815-99e1

By Barry Burger, Innovations Chemist

- Determine alcohols at 0.1-12 mass percent.
- Determine ethers at 0.1-20 mass percent.
- Separate all oxygenates in 11 minutes.

American Society for Testing and Materials Method D-4815-99 is optimized for determining oxygenated additives — ethers and alcohols — in gasolines. Concentrations of individual alcohols can be determined at between 0.1 and 12 mass percent; individual ethers can be determined between 0.1 and 20 mass percent. The chromatographic system consists of two columns, connected via a ten-port gas sampling valve (Figure 1). Column 1 is a Silcosteel® treated micropacked column containing highly polar 1,2,3-tri-2-cyanoethoxypropane (TCEP). Column 2 is a capillary column containing a non-polar polydimethylsiloxane (PDMS) polymer bonded in either fused silica tubing (Rtx®-1 column) or, for greater durability, but equivalent inertness, Silcosteel® treated stainless steel tubing (MXT®-1 column).

The sample is introduced onto the TCEP column. The lighter hydrocarbons quickly pass through this column and are vented, but the heavier hydrocarbons and oxygenates are retained. After methylcyclopentane is eluted, but before di-isopropyl ether (DIPE) and MTBE are eluted, the valve is actuated, backflushing the oxygenates and heavy hydrocarbons onto the PDMS column. From here, the alcohols and ethers elute in boiling point order (Figure 2). After benzene and TAME are eluted, the valve is actuated again, to backflush and vent the heavy hydrocarbons. To prevent adsorption of oxygenates in the sample pathway, and ensure symmetric peaks, we use Siltek®/Sulfinert® treated stainless steel transfer lines and a Sulfinert® treated sampling valve.

This procedure is a fast, reliable means of quantifying the oxygenated compounds currently added to gasolines. Sulfinert® treated system components help ensure accurate data and peak symmetry.

**Figure 1** Inert two-column system for analyzing oxygenates in gasolines.

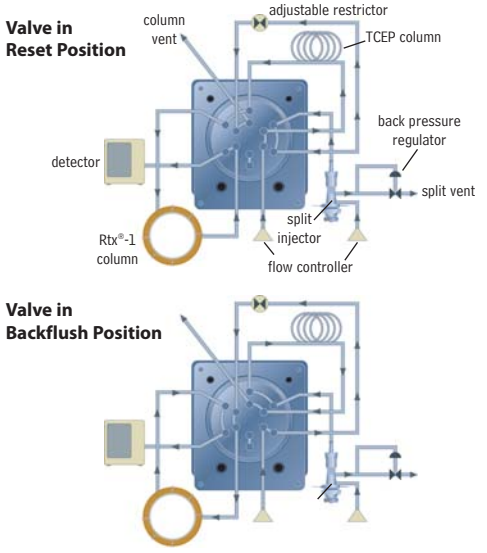
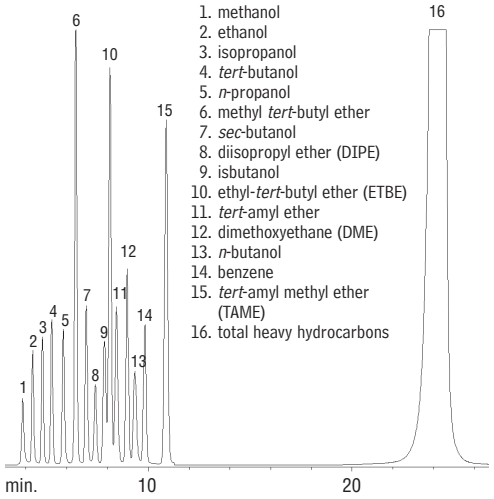



**Figure 2** A TCEP / Rtx®-1 column pair resolves oxygenates added to gasolines.




GC_PC00194

0.56m, 0.75mm ID 20% TCEP on Chromosorb® PAW (cat.# 19040) and 30m, 0.53mm ID, 3.0µm Rtx®-1 (cat.# 10185 connected in series)
0.5µL split injection of oxygenates blend 1–10% wt in surrogate gasoline

| | |
|---|---|
| Oven temp.: | 60°C |
| Inj. / det. temp.: | 200°C / 250°C (FID) |
| Carrier gas: | helium, 5mL/min. set @ 60°C |
| Split ratio: | 15:1 |

### Micropacked Column

| Description | ID | OD | temp. range | length | cat. # |
|---|---|---|---|---|---|
| 20% TCEP on 80/100 Chromosorb® PAW | 0.75 | 1/16" | 0–120°C | 0.56-Meter | 19040 |

### Rtx®-1 Column (fused silica) (Crossbond® 100% dimethyl polysiloxane)

| ID | df (µm) | temp. limits | length | cat. # |
|---|---|---|---|---|
| 0.53mm | 3.00 | -60 to 270/290°C | 30-Meter | 10185 |

### MXT®-1 Column (Silcosteel® treated stainless steel)

(Crossbond® 100% dimethyl polysiloxane)

| ID | df (µm) | temp. limits | length | cat. # |
|---|---|---|---|---|
| 0.53mm | 3.00 | -60 to 285°C | 30-Meter | 70185 |

### Sulfinert® Treated Ten-Port Gas Sampling Valve

| Description | qty. | cat.# |
|---|---|---|
| Sulfinert® Gas Sampling Valve; 10-Port | ea. | 20586 |

### Siltek®/Sulfinert® Treated Coiled 304 Grade Stainless Steel Tubing

| ID | OD | cat.# | 5-24 ft. | 25-199 ft. | 200-399 ft. | >400 ft. |
|---|---|---|---|---|---|---|
| 0.020" (0.51mm) | 1/16" (1.59mm) | 22503 | | | | |

### California Oxygenates Mix

| | | | | | |
|---|---|---|---|---|---|
| diisopropyl ether | 2,000 | *tert*-amyl methyl ether | 2,000 | methyl *tert*-butyl ether | 2,000 |
| ethyl-*tert*-butyl ether | 2,000 | *tert*-butyl alcohol | 10,000 | | |

At indicated concentrations (µg/mL) in P&T methanol, 1mL/ampul
cat. # 30465 (ea.)

# 12-Minute GC Analysis for 33 Organochlorine Pesticides

## Using an Rtx®-440 / Rtx®-CLPesticides2 Column Pair

By Jason Thomas, Environmental Innovations Chemist

- High sample throughput.
- Excellent resolution and responses.
- Equivalent column lifetimes.

A ubiquitous and persistent presence in the environment, and possibly carcinogenic properties, make organochlorine pesticides one of the most commonly analyzed groups of compounds in environmental assays. Because of this prevalence, it is important to use columns that can 1) separate these numerous and varying compounds, to ensure accurate data, and 2) provide short analysis times, to ensure adequate throughput for the laboratory. Further, the columns must be compatible with analytical conditions that ensure good response for these active, difficult-to-analyze compounds. An Rtx®-440 / Rtx®-CLPesticides2 column pair fulfills these requirements.

To illustrate the capabilities of these columns, we chose the 33 organochlorine pesticides in the target list for Revision 1 of US EPA Method 8081A. Several of these compounds are described as possibly co-eluting pairs on the columns listed in the method, but all are resolved on the unique Rtx®-440 stationary phase. A combination of a 30m x 0.32mm ID x 0.5µm Rtx®-440 column and an Rtx®-CLPesticides2 column in the same configuration (cat. # 12939 and cat.# 11325, repectively), is an excellent choice for the analysis.

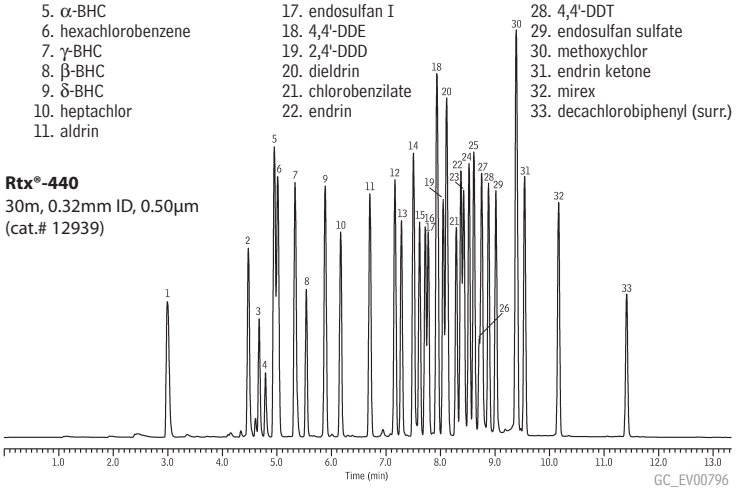
Figure 1 shows all 33 compounds elute in 12 minutes, allowing high throughput. Splitless injection at 275°C, with a 0.75 minute hold time and using an inert Siltek® treated splitless single gooseneck inlet liner (cat.# 20961-214.1), assures good responses. Oven temperature conditions are optimized to allow excellent resolution, quickly, by the Rtx®-440 phase. Because conditions are identical for the two analyses, and analysis times are equal, an efficient way to monitor these pesticides is by splitting a single injected sample to the two columns, and two detectors. This eliminates the need for a separate confirmation run, or for GC/MS analysis, and can markedly improve productivity.

The new 0.5µm stationary phase thickness makes the 30m x 0.32mm ID x 0.5µm Rtx®-CLPesticides2 column compatible with the 30m x 0.32mm ID x 0.5µm Rtx®-440 column, or with other pesticide columns of like phase ratio, eliminating disparity in potential column life expectancies. For accurate, high throughput analyses of organochlorine pesticides, we highly recommend this Rtx®-440/Rtx®-CLPesticides2 column pair.

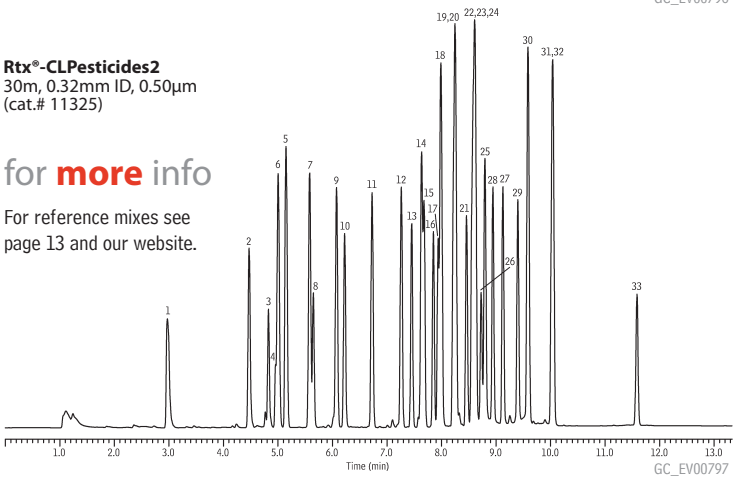
**Figure 1** Separate 33 organochlorine pesticides in 12 minutes, using conditions optimized for an Rtx®-440 column.

1. hexachlorocyclopentadiene
2. 2,4,5,6-tetrachloro-*m*-xylene (surr.)
3. *cis*-diallate
4. *trans*-diallate
5. α-BHC
6. hexachlorobenzene
7. γ-BHC
8. β-BHC
9. δ-BHC
10. heptachlor
11. aldrin
12. isodrin
13. heptachlor epoxide
14. 2,4'-DDE
15. γ-chlordane
16. α-chlordane
17. endosulfan I
18. 4,4'-DDE
19. 2,4'-DDD
20. dieldrin
21. chlorobenzilate
22. endrin
23. 2,4'-DDT
24. 4,4'-DDD
25. endosulfan II
26. kepone
27. endrin aldehyde
28. 4,4'-DDT
29. endosulfan sulfate
30. methoxychlor
31. endrin ketone
32. mirex
33. decachlorobiphenyl (surr.)

**Rtx®-440**
30m, 0.32mm ID, 0.50µm
(cat.# 12939)




**Rtx®-CLPesticides2**
30m, 0.32mm ID, 0.50µm
(cat.# 11325)




for **more** info

For reference mixes see page 13 and our website.

| | |
|---|---|
| Sample: | Organochlorine Pesticides Mix AB #2 (cat.# 32292), Chlorobenzilate (cat.# 32211) 1000µg/mL in methanol, Diallate (*cis* & *trans*) (custom) 1000µg/mL in hexane, Hexachlorobenzene (cat.# 32231) Hexachlorocyclopentadiene (cat.# 32232), Isodrin (custom) 1000µg/mL in hexane, Kepone (custom) 1000µg/mL in hexane, Mirex (custom) 1000µg/mL in hexane, 2,4'-DDD (cat.# 32098), 2,4'-DDE (cat.# 32099), 2,4'-DDT (cat.#32200), TCMX (cat.# 32027), DCB (cat.# 32029) |
| Inj.: | 1.0µL splitless (hold 0.75 min.), 2mm Siltek® treated single gooseneck inlet liner (cat.# 20961-214.1). Sample divided to two columns, using Siltek® treated 0.32mm ID tubing (cat.# 10027) and a SeCure™ "Y" connector (cat.# 20277). |
| Inj. temp.: | 275°C |
| Carrier gas: | hydrogen, constant pressure |
| Linear velocity: | 51 cm/sec. @ 140°C |
| Oven temp.: | 140°C (hold 1 min.) to 240°C @ 30°C/min. (hold 2 min.) to 330°C @ 30°C/min. (hold 4 min.) |
| Det.: | ECD @ 330°C |
| Instrument: | Shimadzu GC-2010 with ECD and FID |

### Rtx®-CLPesticides2 Column (fused silica)

| ID | df (µm) | temp. limits | length | cat. # |
|---|---|---|---|---|
| 0.32mm | 0.50 | -60 to 320/340°C | 30-Meter | 11325 |

### Rtx®-440 Column (fused silica)

| ID | df (µm) | temp. limits | length | cat. # |
|---|---|---|---|---|
| 0.32mm | 0.50 | 20°C to 320/340°C | 30-Meter | 12939 |

# 0.5pg Limit of Detection for Cocaine

## Using an Allure™ PFP Propyl Column and HPLC/TOF-MS

By Kristi Sellers, Clinical/Forensics Innovations Chemist

- Monitor cocaine and benzoylecgonine at 0.5pg on-column, ecgonine methylester at 5pg.
- Analysis completed in less than 3 minutes.
- Fast, simple sample preparation—no need for derivatization.

When cocaine is introduced into the body, several main metabolites are produced: benzoylecgonine, ecgonine, and ecgonine methylester. To determine the presence of cocaine and/or these metabolites, urine samples are screened using enzyme immunoassay, and positive results are confirmed using GC/MS. Although GC/MS methods are well established, and provide excellent confirmation data, they can be time-consuming, due to multiple sample preparation steps, including derivatization — and long analysis times.[1] HPLC coupled with electrospray (ESI) time-of-flight mass spectrometry provides an alternate chromatographic confirmation method for cocaine and its metabolites. Using an Allure™ PFP Propyl column in combination with a high-organic mobile phase provides short analysis times and allows detection limits at low picogram levels, without derivatization.

Cocaine, benzoylecgonine, and ecgonine methylester are hydrophilic, basic drugs with $pK_a$ values greater than 8. Consequently, buffer salts or ion-pairing agents and a low-organic mobile phase are needed to ensure adequate retention on a typical C18 reversed phase column. Some retention can be achieved under these conditions, but the highly aqueous mobile phase causes poor MS response due to inefficient desolvation, and the salts cause ion suppression during ESI.[2] Under optimal screening conditions, limits of detection of 1ng/mL for cocaine and 5ng/mL for benzoylecgonine have been reported (10pg and 50pg on-column, respectively; 10µL injection).[3]

In contrast, the combination of an Allure™ PFP Propyl column and a high-organic mobile phase provides not only adequate retention and short analysis times, but also excellent sensitivity. All target compounds are eluted from the 30mm column within 3 minutes (Figure 1), with reliable reproducibility of responses (Table 1). S/N:RMS values greater than 90 indicate excellent sensitivity at 5.0pg on-column for all compounds; values of 16 and greater indicate adequate sensitivity for most compounds at 0.5pg on-column. For each compound the relative standard deviation (%RSD) for intensity is below 10% across a broad concentration range, except for the 0.5pg value for metabolite ecgonine methylester (Table 1).

Because the Allure™ PFP Propyl column and the high-organic mobile phase, coupled with HPLC/TOF-MS, produce highly reproducible signal intensities for each mass down to 5pg on-column, we recommend this column and these analysis parameters as an alternative chromatographic approach for confirmation of cocaine and its metabolites.

**References**

1. Jeanville, P.M., E.S. Estape, S.R. Needham, M.J. Cole, J. Am. Soc. Mass Spectrom, 11: 257-263 (2000).
2. Needham, S.R., P.M. Jeanville, P.R. Brown, E.S. Estape, J. Chromatography B, 748: 77-87 (2000).
3. Milner, C., R. Kinghorn, *Development of a Screening Analysis by LC Time-of-Flight MS for Drugs of Abuse*, Agilent Technologies, publication 5989-3157EN www.agilent.com/chem

### Allure™ PFP Propyl Column

| 5µm Column, 2.1mm | cat. # |
|---|---|
| 30mm | 9169532 |

**Figure 1** Rapid, sensitive analysis for cocaine and its metabolites, using an Allure™ PFP Propyl column


| Peak List | Mass | Ret. Time (min.) |
|---|---|---|
| 1 ecgonine methylester | 200 | 1.27 |
| 2 benzoylecgonine | 290 | 2.00 |
| 3 cocaine | 304 | 2.33 |

**Sample:**
Inj.: 10µL, 5pg each compound on column
Solvent: mobile phase

**Column:** Allure™ PFP Propyl
Cat.#: 9169532
Dimensions: 30mm x 2.1mm
Particle size: 5µm
Pore size: 60Å

**Conditions:**
Mobile phase: acetonitrile:5mM ammonium formate in water, pH=3.0, 80:20
Flow: 0.6mL/min.
Temp.: 25°C

**Detection:** MS, Micromass LCT Premier
ESI: positive
Capillary: 3000V
Sample cone: 20V
Desolvation temp.: 250°C
Source temp.: 120°C

**Table 1** Reproducible responses for cocaine and its metabolites across a wide range of concentrations.

| On-column Amount (ng) | Mean % RSD (n = 7) COC | BZE | EME | Cd3 |
|---|---|---|---|---|
| 250 | 1.9 | 3.2 | 1.5 | 3.2 |
| 125 | 0.4 | 4.7 | 1.5 | 4.0 |
| 25 | 0.5 | 4.8 | 0.6 | 4.4 |
| 5 | 0.8 | 2.9 | 1.5 | 2.1 |
| 2.5 | 1.7 | 4.2 | 3.2 | 2.4 |
| 0.5 | 5.7 | 0.7 | 0.9 | 2.8 |
| 5pg | 7.4 | 8.1 | 7.8 | 5.1 |
| 0.5pg | 4.3 | 8.0 | 40.7 | 6.3 |

COC - cocaine; BZE - benzoylecgonine; EME - ecgonine methylester; Cd3 - cocaine-d3

# Evaluating Peppermint Oils by Chiral GC/MS

## Using an Rt-γDEXsa™ Column

By Julie Kowalski, Innovations Chemist

- Rt-γDEXsa™ column has unmatched selectivity for peppermint oil stereoisomers.
- GC/MS enables detailed comparisons of natural products.
- Analysis requires no sample preparation.

Peppermint has a long history as a flavor additive and herbal remedy. Ancient Greeks and Romans adorned themselves with peppermint leaves and used the oil to flavor sauces and wines.[1] Today, peppermint is widely used in foods, candies, alcoholic and non-alcoholic beverages, cosmetics, personal care products, perfumes and, of course, chewing gum. Modern research has demonstrated health benefits of peppermint include antispasmodic, carminative, cholagogue, antibacterial, secretolytic, and cooling activity.[2]

Peppermint oil is isolated from the plant *Mentha piperita*. Preparation begins with harvesting leaves and flowering tops from the plant. Although other techniques exist, steam distillation is the most commonly used method for extracting highly concentrated oil from the plant material.

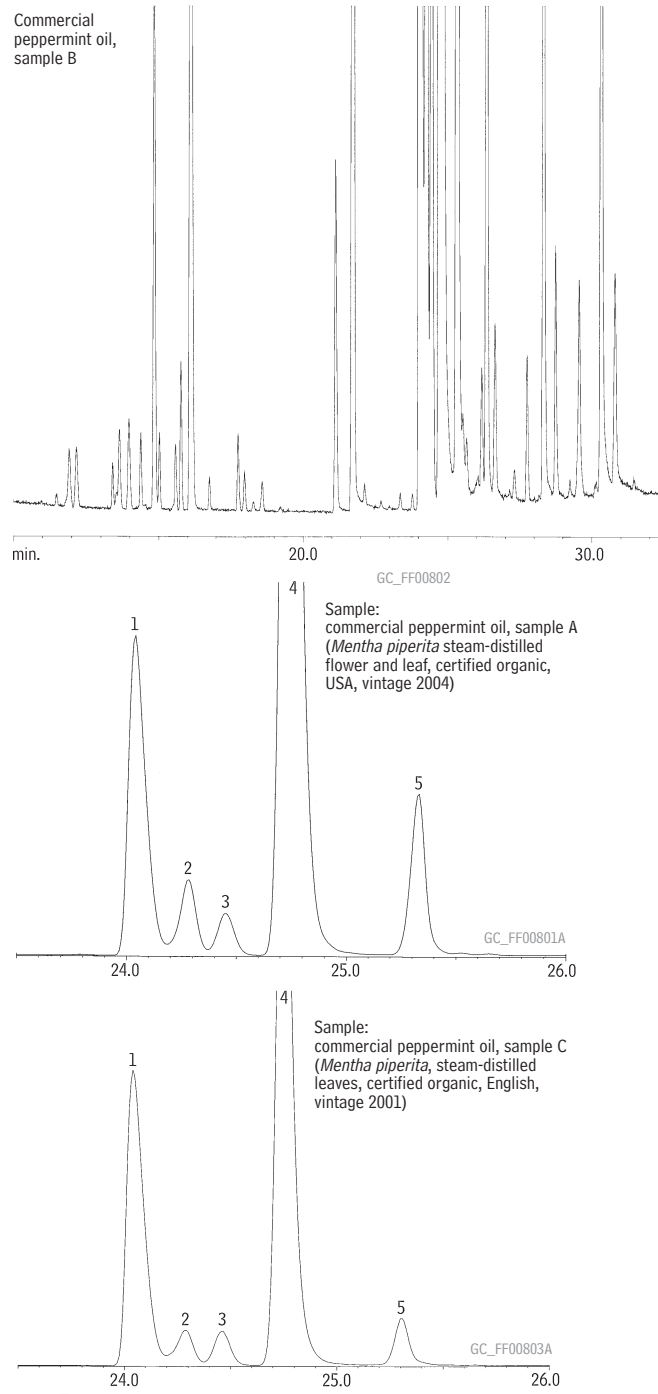
The use of peppermint oil in commercial goods and the rising demand for all-natural products drive a need for more rigorous testing. Often, a product is adulterated to increase desirable properties of the natural oil or to avoid costly manufacturing of all-natural oil. Adulteration usually is accomplished by adding a similar but cheaper oil, such as cornmint oil (*Mentha arvensis*), or by diluting the oil with various solvent oils. Adulteration and quality consistency of peppermint oil fuels concern over compromised quality, but also introduces health safety issues; for example, there is potential for an allergic reaction to an added unnatural compound or excess of a natural component.

Reliable, sensitive analytical methods are vital to detect complex manipulation of synthetic materials to mimic natural materials. Also, standardizing the composition that defines a "natural" product can be difficult, due to natural variation among plants and plant varieties, variation in geographical and seasonal factors, and inconsistencies in the manufacturing process. Some naturally occurring stereoisomers show greater biological activity than their counterparts: (-)-menthol, for example, is the stereoisomer known to have the greatest cooling and scent effects. This inequality between stereoisomers can be used to advantage, because reliable methods of analyzing specific chiral components can be used to monitor product quality.

Despite the value of identifying and quantifying major components like menthol, methone, and methyl acetate, dependable identification and quantification is difficult because each of these is represented by several stereoisomers. Menthol, for example, has three chiral centers, for a total of eight stereoisomers, making chromatographic separation difficult.

Here, we show a robust chiral GC/MS method that can be used with confidence to characterize and quantify stereoisomeric compounds. We purchased four peppermint oil samples from four commercial sources. Each sample was identified by information provided at time of purchase, including harvest location and year. Analyses were performed in triplicate on a Shimadzu GC/MS; model GC-17A, MS-QP5000, using an Rt-γDEXsa™ cyclodextrin-based column. The autosampler program included extensive rinsing with methylene chloride to prevent sample memory and syringe plugging. Data were analyzed using Shimadzu LabSolution, Version 1.20. Identifications of

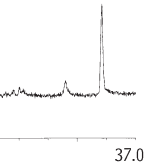
**Figure 1** A representative commercial peppermint oil (sam
oils that differ in harvest location and year, using an Rt-γDE

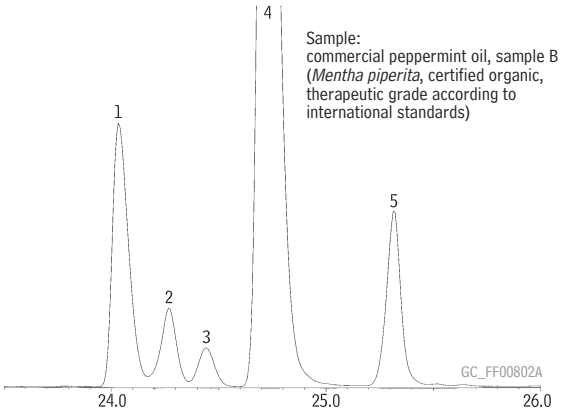


ple B), and major chiral components of four commercial
Xsa™ column.

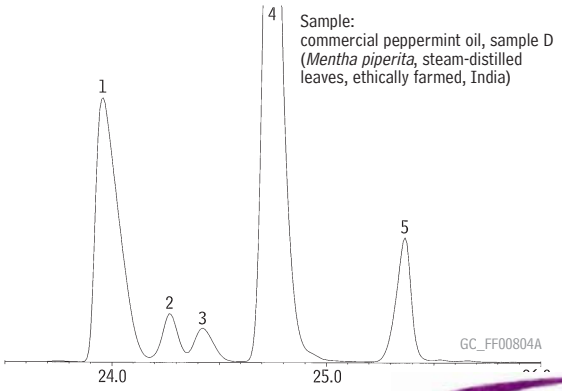
| | |
|---|---|
| Column: | Rt-γDEXsa™ 30m x 0.25mm ID, 0.25μm (cat.# 13113) |
| Inj.: | 1.0μL neat, split (split ratio 1:150) |
| Inj. temp.: | 230°C |
| Carrier gas: | helium, constant pressure |
| Flow rate: | 35cm/sec. at 100°C |
| Oven temp.: | 40°C to 120°C @ 5°C/min. to 135°C @ 3°C/min. to 200°C @ 5°C/min. |
| Det: | MS |
| Transfer line temp.: | 200°C |
| Scan range: | 40-300amu |
| Ionization: | EI |
| Mode: | scan |

1. menthone
2. menthol
3. menthone
4. menthol
5. menthyl acetate




Sample:
commercial peppermint oil, sample B
(*Mentha piperita*, certified organic, therapeutic grade according to international standards)




Sample:
commercial peppermint oil, sample D
(*Mentha piperita*, steam-distilled leaves, ethically farmed, India)

menthol, methone, and methyl acetate isomers were based on retention time comparisons to literature values and a mass spectra library search.[3] Integration of chromatograms was consistent. Additional menthol isomers were detected, but chromatographic conditions were optimized for those in Figure 1.

The figures show the Rt-γDEXsa™ phase is well suited to separating the stereoisomers of the major chiral components, and enables the analyst to differentiate among peppermint oils from different sources, and between peppermint oil and cornmint oil.* These chromatograms are similar, despite differences in harvest location, as illustrated by commercial samples A, C, and D. Similarity extends to samples which were harvested in different years, as shown by commercial samples A and C.

The Rt-γDEXsa™ phase allows detection of major components important to the quality of peppermint oil product, thus providing manufacturers and buyers with consistent profiles with which to confirm and track product quality. We offer a broad range of cyclodextrin-based chiral columns for analyzing oils or other enantiomer-containing materials. These columns are available individually or in kits that can help you select the best column, or combination of columns, for a particular application. In addition, our experienced Technical Service and Innovations chemists are always ready to help you resolve concerns about your particular chiral analysis.

**References**
1. http://www.botanical.com/botanical/mgmh/m/mints-39.html#pep)
2. http://www.umm.edu/altmed/ConsHerbs/Peppermintch.html
3. J. Chromatogr. A, 1054: 87-93 (2004).

## Rt-γDEXsa™ Columns (fused silica)

(2,3-di-acetoxy-6-O-*tert*-butyl dimethylsilyl gamma cyclodextrin doped into 14% cyanopropylphenyl/86% dimethyl polysiloxane)

| ID | df (μm) | temp. limits | length | cat. # |
|---|---|---|---|---|
| 0.25mm | 0.25 | 40 to 230°C | 30-Meter | 13113 |
| 0.32mm | 0.25 | 40 to 230°C | 30-Meter | 13112 |

## Essential Oils Chiral Column Kits (fused silica)

| Dimensions & Columns | cat.# |
|---|---|
| 30m, 0.25mm ID, 0.25μm<br>Rt-ßDEXsm™, Rt-ßDEXse™, Rt-ßDEXsa™, & Rt-ßDEXsp™ columns | 13196 |
| 30m, 0.32mm ID, 0.25μm<br>Rt-ßDEXsm™, Rt-ßDEXse™, Rt-ßDEXsa™, & Rt-ßDEXsp™ columns | 13197 |

For many other chiral columns and kits, refer to our catalog, or visit our website.

## for **more** info

*For a chiral analysis of cornmint oil, visit our website (search: GC_FF00805).

# Comprehensive Dual-Column GC for Pharmaceutical Solvents

## Analyze all EPA Method 1671 Analytes, Using a Stabilwax® / Stabilwax® DB Column Pair

By Rick Lake, Pharmaceutical Applications Chemist

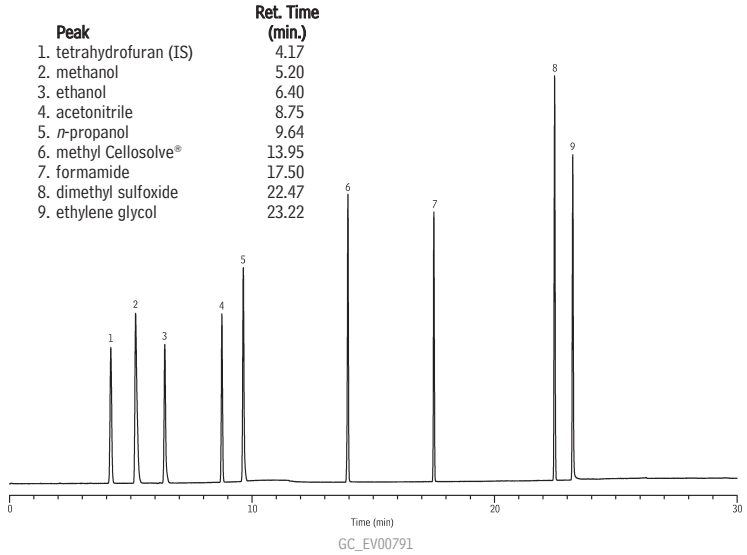
- Dual-column approach targets all listed potential pollutants with one injection.
- Excellent resolution and peak shapes from two polyethylene glycol (PEG) stationary phases.
- Superior analysis of the primary amines from a base-deactivated PEG.

The fate of solvents used in manufacturing pharmaceutical products is a worldwide concern. In the US, EPA Method 1671, *Volatile Organic Compounds Specific to the Pharmaceutical Manufacturing Industry by GC/FID* is used to monitor concentrations of non-purgeable pollutants in the aqueous discharge of pharmaceutical manufacturing facilities. Method 1671 is a performance-based method and, therefore, may be tailored to specific analytes and advantageous techniques, if all acceptance criteria are upheld. The target analytes—water-soluble organic solvents used in manufacturing pharmaceutical products—include primary amines (methyl amine, dimethylamine, diethylamine, triethylamine), which are basic in nature, and alcohols (methanol, ethanol, ethylene glycol), which are slightly acidic. When the diverse chemical properties of the target analytes are considered, column selection might be the most important aspect to developing a successful approach to this method.

The first step in developing any GC method is selection of the stationary phase. The diverse chemical nature of the analytes in Method 1671 is a major consideration. Polyethylene glycol (PEG) stationary phases are an excellent choice for analyzing organic solvents in a water matrix. PEG is a polar material with the capacity to resolve all applicable analytes, as well as to retain polar solvents, like water. Retention of polar solvents is advantageous here, because it enables the analyst to inject larger samples without the worry of extinguishing the FID. Yet, because both basic and acidic properties are represented by the analytes, no single analytical column may be capable of effectively analyzing all analytes: a column incorporating a base-deactivated functionality might act against the chromatography of the acidic alcohols and, likewise, a column without specific base deactivation might not provide acceptable chromatography for the basic primary amines. Therefore, we used a dual-column method comprising both base-deactivated and non-base-deactivated PEG stationary phases to analyze the entire Method 1671 target list.

Using a SeCure™ "Y" Connector (cat.# 20278), we divided the flow leaving the inlet of the GC system into two 30m, 0.32mm, 1.0µm columns: a Stabilwax® column and a Stabilwax® DB column

**Figure 1** Excellent resolution and peak symmetry for non-basic pharmaceutical solvents, using a Stabilwax® column.

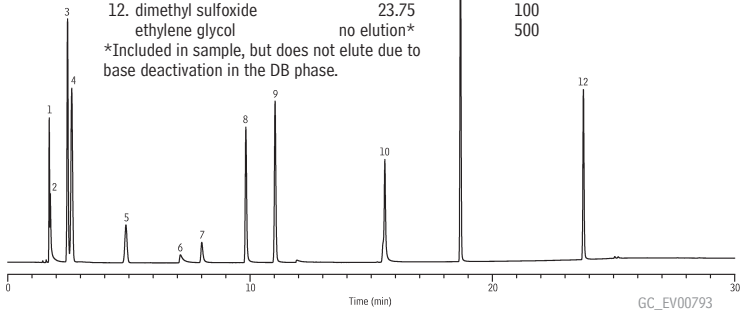



| | |
|---|---|
| Columns: | shown: Stabilwax® 30m, 0.32mm ID, 1.0µm (cat.# 10654)<br>(Column 2: Stabilwax® DB 30m, 0.32mm ID, 1.0µm (cat.# 10854))<br>Flow from injector split to two columns using 0.53mm ID intermediate-polarity deactivated guard column (cat.# 10045), SeCure™ "Y" Connector (cat.# 20278), and "Y" Press-Tight® Connector (cat.# 20405) |
| Sample: | 1671 volatile organics mix in deionized water, 100µg/mL each compound |
| Inj.: | 1.0µL, split (split ratio 12:1), 4mm gooseneck splitless inlet liner (cat.# 20798) |
| Inj. temp.: | 200°C |
| Carrier gas: | helium, constant pressure |
| Linear velocity: | 2.48mL/min. / 39.25cm/sec @ 40°C<br>(Column 2: 2.51mL/min. / 39.68cm/sec @ 40°C) |
| Oven temp.: | 40°C (5 min.) to 180°C @ 7°C/min., hold 5 min. |
| Det.: | FID @ 250°C |

**Figure 2** A Stabilwax® DB column provides excellent chromatography for basic and non-acidic pharmaceutical solvents.

| Peak | Retention Time (min.) | Concentration (µg/mL) |
|---|---|---|
| 1. dimethylamine | 1.71 | 200 |
| 2. methylamine | 1.76 | 200 |
| 3. diethylamine | 2.46 | 200 |
| 4. triethylamine | 2.64 | 200 |
| 5. tetrahydrofuran (IS) | 4.88 | 100 |
| 6. methanol | 7.12 | 40 |
| 7. ethanol | 8.01 | 40 |
| 8. acetonitrile | 9.82 | 200 |
| 9. *n*-propanol | 11.03 | 200 |
| 10. methyl Cellosolve® | 15.56 | 200 |
| 11. formamide | 18.68 | 500 |
| 12. dimethyl sulfoxide | 23.75 | 100 |
| ethylene glycol | no elution* | 500 |

*Included in sample, but does not elute due to base deactivation in the DB phase.




| | |
|---|---|
| Columns: | shown: Stabilwax® DB 30m, 0.32mm ID, 1.0µm (cat.# 10854)<br>(Column 1: Stabilwax® 30m, 0.32mm ID, 1.0µm (cat.# 10654))<br>Flow from injector split to two columns using 0.53mm ID intermediate-polarity deactivated guard column (cat.# 10045), SeCure™ "Y" Connector (cat.# 20278), and "Y" Press-Tight® Connector (cat.# 20405) |
| Sample: | 1671 volatile organics mix in deionized water, concentrations indicated on figure. |
| Inj.: | 1.0µL, split (split ratio 12:1), 4mm gooseneck splitless inlet liner (cat.# 20798) |
| Inj. temp.: | 200°C |
| Carrier gas: | helium, constant pressure |
| Linear velocity: | 2.51mL/min. / 39.68cm/sec @ 40°C (Column 1: 2.48mL/min. / 39.25cm/sec @ 40°C) |
| Oven temp.: | 40°C (5 min.) to 180°C @ 7°C/min., hold 5 min. |
| Det.: | FID @ 250°C |

(cat.# 10654 and cat.# 10854, respectively). Rather than using a splitless injection, as listed in the method, we used a split injection at a 12:1 ratio to enhance peak shape for the analytes. By retaining the water solvent, as described above, the polar PEG stationary phases enabled us to increase the injection volume to 1.0µL. As a result, we achieved the detection limits specified in the method, using a split injection.

The Stabilwax® column demonstrated excellent separation and peak shape for the non-basic analytes (Figure 1). Had the target compounds not included primary amines, this column alone would be a good choice. The amines broke down almost totally on the Stabilwax® column, however. Conversely, the Stabilwax® DB column, which is designed for analyses of basic compounds, exhibited excellent resolution and peak shape for the amines and non-acidic analytes, but produced excessive tailing of the methanol and ethanol peaks, and nearly complete breakdown of ethylene glycol (Figure 2). By selecting the appropriate information from this Stabilwax®/Stabilwax® DB dual-column GC/FID analysis, a comprehensive picture for all Method 1671 analytes, both acidic and basic, can be obtained.

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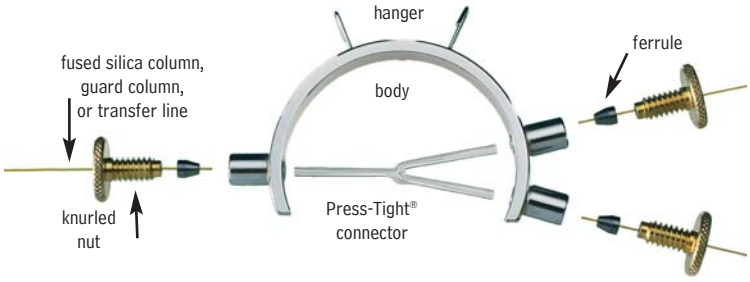



**The SeCure™ "Y" connector's open design allows visual confirmation of the seal; secondary seals ensure a leak-tight connection.**

- Connect two analytical columns to a transfer line or guard column.
- Use standard "Y" Press-Tight® connectors and 1/16" graphite ferrules.
- Reliable seal integrity, will not unexpectedly disconnect during temperature-programmed analyses.
- Open design allows visual confirmation of the seal for added confidence in the connection.

Combine the simplicity of a "Y" Press-Tight® connector with the strength of a metal union. The ferrules and knurled nuts hold the fused silica tubing in place, which prevents the tubing from unexpectedly disconnecting, even at temperatures as high as 400°C.

## Stabilwax® Column (fused silica)

(Crossbond® Carbowax® polyethylene glycol)

| ID | df (µm) | temp. limits | length | cat. # |
|---|---|---|---|---|
| 0.32mm | 1.00 | 40 to 240/250°C | 30-Meter | 10654 |

## Stabilwax®-DB Column (fused silica)

(Crossbond® Carbowax® polyethylene glycol for amines and basic compounds)

| ID | df (µm) | temp. limits | length | cat. # |
|---|---|---|---|---|
| 0.32mm | 1.00 | 40 to 210/220°C | 30-Meter | 10854 |

## Universal "Y" Press-Tight® Connectors

| Description | cat. # |
|---|---|
| Universal "Y" Press-Tight® Connector | 20405 |
| Deactivated Universal "Y" Press-Tight® Connector | 20405-261 |
| Siltek® treated Universal "Y" Press-Tight® Connector | 20485 |

## SeCure™ "Y" Connector Kits

| Description | Ferrules Fit Column ID | qty. | cat.# |
|---|---|---|---|
| SeCure™ "Y" Connector | 0.25/0.28mm | kit | 20276 |
| SeCure™ "Y" Connector | 0.32mm | kit | 20277 |
| SeCure™ "Y" Connector | 0.45/0.53mm | kit | 20278 |
| Knurled nut | | 3-pk. | 20279 |

Kits include: SeCure™ "Y" connector body, 3 knurled nuts, "Y" Universal Press-Tight® union, 3 ferrules.

# Sulfinert®-Treated Sample Cylinders

## Increase Storage Time for Active Sulfur Compounds

By Neil Mosesman, Air Monitoring Product Marketing Manager

Stainless steel sample cylinders commonly are used in the collection and analysis of refinery and natural gas samples. These samples often contain trace amounts of sulfur-containing compounds (e.g., hydrogen sulfide, mercaptans, and sulfides) which can interfere with reactions or poison catalysts in petrochemical processes. Because sulfur compounds quickly react with stainless steel surfaces, accurate determination of these compounds is impossible when using untreated sample cylinders.

Restek's proprietary Sulfinert® passivation technique bonds an inert silica layer into the surface of the stainless steel, preventing active compounds from reacting with or adsorbing to the stainless steel. Therefore, Sulfinert® products are ideal for storing and transferring reactive sulfur compounds.

Most stainless steel system components, including valves, sample loops, and tubing, can be treated with Sulfinert® passivation (e.g., see page 5). Because the Sulfinert® layer is incorporated into the structure of the stainless steel, treated surfaces can be bent or flexed without affecting their inertness characteristics.

As shown in Figure 1, Sulfinert®-treated cylinders and accessories are inert to reactive sulfur compounds. Hydrogen sulfide exhibited greater than 85% recovery over the test period; methyl mercaptan, ethyl mercaptan, carbonyl sulfide, and dimethyl disulfide exhibited greater than 90% recovery.

Sulfinert®-treated gas sampling equipment is ideal for collecting and storing samples containing ppb levels of sulfur compounds, such as natural gas or beverage-grade carbon dioxide. Sulfinert® treatment ensures that sulfur compounds or other highly active compounds remain stable during transport from the field to the laboratory.

### Sulfinert®-Treated Swagelok® Sample Cylinders

- Stable storage of samples containing ppb levels of sulfur compounds.
- D.O.T. rated to 1800psi at room temperature.
- High quality cylinders manufactured by Swagelok®.

| Size | qty. | cat.# |
|---|---|---|
| 75cc | ea. | 24130 |
| 150cc | ea. | 24131 |
| 300cc | ea. | 24132 |
| 500cc | ea. | 24133 |
| 1000cc | ea. | 24134 |
| 2250cc | ea. | 21394 |

### Sulfinert®-Treated Alta-Robbins Sample Cylinder Valves

- All wetted parts are Sulfinert®-treated for inertness.
- Compatible with Sulfinert®-treated Swagelok® sample cylinders.
- Large, durable, Kel-F® seat ensures leak-free operation.

| Description | qty. | cat.# |
|---|---|---|
| 1/4" NPT Exit | ea. | 21400 |
| 1/4" Compression Exit | ea. | 21401 |
| 1/4" NPT with Dip Tube* | ea. | 21402 |
| 1/4" NPT with 2850psi Rupture Disk | ea. | 21403 |

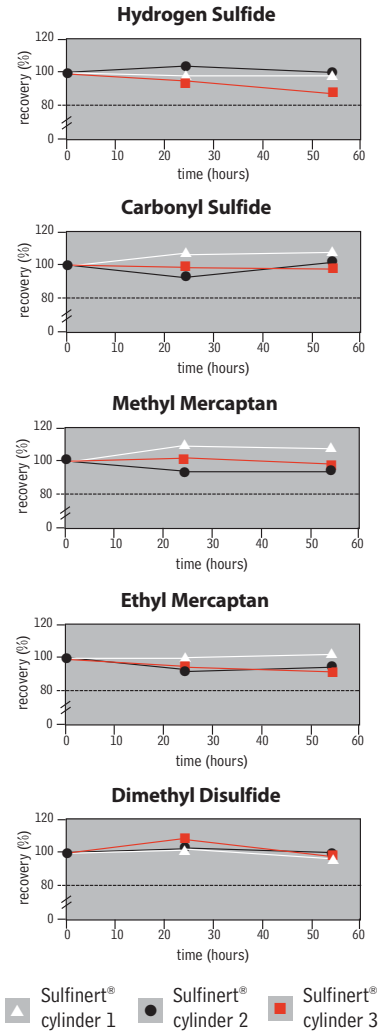
*Specify dip tube length or % outage when ordering (maximum length = 5.25"/ 13.3cm)

### Sulfinert®-Treated Rupture Disc Tee

850psig rating; 1/4" NPT connections.

| Description | qty. | cat.# |
|---|---|---|
| Sulfinert®-Treated Rupture Disc Tee (1/4" NPT connections) | ea. | 21396 |
| Replacement Rupture Disc (not Sulfinert®-treated) | ea. | 24298 |

**Figure 1** Stability of sulfur compounds is remarkable in Sulfinert®-treated cylinders.

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**Sulfinert® treated sampling apparatus.**

Sample Cylinder (cat. # 24133)

Cylinder Valve (cat. # 21400)

Rupture Disc Tee (cat. # 21396)

For Sulfinert® treated fittings, tubing, and sample loops, refer to our catalog or visit our website.

# Analytical Reference Materials

Semivolatile Internal Standards & Surrogates; Organochlorine Pesticides; Oxygenates

by Ken Herwehe, Analytical Reference Materials Product Marketing Manager

## Semivolatile Internal Standards & Surrogates

### Revised SV Internal Standard Mix (7 components)

acenaphthane-d10 — naphthalene-d8
chrysene-d12 — perylene-d12
1,4-dichlorobenzene-d4 — phenanthrene-d10
1,4-dioxane-d8

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31885 (ea.)

4,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31886 (ea.)

### Revised B/N Surrogate Mix

2-fluorobiphenyl — *p*-terphenyl-d14
nitrobenzene-d5 — pyrene-d10

1,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31887 (ea.)

5,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31888 (ea.)

5,000µg/mL each in methylene chloride, 5mL/ampul
cat. # 31889 (ea.)

### SV Internal Standard Mix

acenaphthene-d10 — naphthalene-d8
chrysene-d12 — perylene-d12
1,4-dichlorobenzene-d4 — phenanthrene-d10

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31206 (ea.)

4,000µg/mL each in methylene chloride, 1mL/ampul*
cat. # 31006 (ea.)

*Warm and sonicate before using.

### B/N Surrogate Mix (4/89 SOW)

2-fluorobiphenyl — *p*-terphenyl-d14
nitrobenzene-d5

1,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31024 (ea.)

5,000µg/mL each in methylene chloride, 1mL/ampul*
cat. # 31062 (ea.)

5,000µg/mL each in methylene chloride, 5mL/ampul*
cat. # 31086 (ea.)

*Warm and sonicate before using.

### Acid Surrogate Standard Mix (3/90 SOW)

2-chlorophenol-d4 — phenol-d6
2-fluorophenol — 2,4,6-tribromophenol

1,500µg/mL each in methanol, 1mL/ampul
cat. # 31003 (ea.)

7,500µg/mL each in methanol, 1mL/ampul
cat. # 31073 (ea.)

7,500µg/mL each in methanol, 5mL/ampul
cat. # 31083 (ea.)

### Acid Surrogate Mix (4/89 SOW)

2-fluorophenol — 2,4,6-tribromophenol
phenol-d6

2,000µg/mL each in methanol, 1mL/ampul
cat. # 31025 (ea.)

10,000µg/mL each in methanol, 1mL/ampul
cat. # 31063 (ea.)

10,000µg/mL each in methanol, 5mL/ampul
cat. # 31087 (ea.)

### B/N Surrogate Standard Mix (3/90 SOW)

1,2-dichlorobenzene-d4 — nitrobenzene-d5
2-fluorobiphenyl — *p*-terphenyl-d14

1,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31002 (ea.)

5,000µg/mL each in methylene chloride, 1mL/ampul*
cat. # 31072 (ea.)

5,000µg/mL each in methylene chloride, 5mL/ampul*
cat. # 31082 (ea.)

*Warm and sonicate before using.

### OLC 03.2 SVOA Deuterated Monitoring Compounds (DMC) (16 components)

acenaphthylene-d8 — 4,6-dinitro-methylphenol-d2
anthracene-d10 — fluorene-d10
benzo(a)pyrene-d12 — 4-methylphenol-d8
4-chloroaniline-d4 — nitrobenzene-d5
bis-(2-chloroethyl)ether-d8 — 2-nitrophenol-d4
2-chlorophenol-d4 — 4-nitrophenol-d4
2,4-dichlorophenol-d3 — phenol-d5
dimethylphthalate-d6 — pyrene-d10

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31810 (ea.)

No data pack available.

## Organochlorine Pesticides

See 12-minute analysis on page 6.

### Organochlorine Pesticide Mix AB #2

(20 components)

| | | | |
|---|---|---|---|
| aldrin | 8µg/mL | dieldrin | 16 |
| α-BHC | 8 | endosulfan I | 8 |
| β-BHC | 8 | endosulfan II | 16 |
| δ-BHC | 8 | endosulfan sulfate | 16 |
| γ-BHC (lindane) | 8 | endrin | 16 |
| α-chlordane | 8 | endrin aldehyde | 16 |
| γ-chlordane | 8 | endrin ketone | 16 |
| 4,4'-DDD | 16 | heptachlor | 8 |
| 4,4'-DDE | 16 | heptachlor epoxide (B) | 8 |
| 4,4'-DDT | 16 | methoxychlor | 80 |

In hexane:toluene (1:1), 1mL/ampul
cat. # 32292 (ea.)

### Pesticide Surrogate Mix

decachlorobiphenyl — 2,4,5,6-tetrachloro-*m*-xylene

200µg/mL each in acetone, 1mL/ampul
cat. # 32000 (ea.)

## Oxygenates

See sub-ppb analysis on page 4.

### Methyl *tert*-butyl ether (MTBE)

2,000µg/mL in P&T methanol, 1mL/ampul
cat. # 30402 (ea.)

### *tert*-Butanol-d9 Standard

20,000µg/mL in P&T methanol, 1mL/ampul
cat. # 30618 (ea.)

did you **know**?

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# Inlet Seals for Agilent Instruments

by Donna Lidgett, GC Accessories Product Marketing Manager


**best** choice!

Washerless, leak-tight seals for Agilent GCs

## Dual Vespel® Ring Inlet Seals

- Vespel® ring embedded in bottom surface eliminates need for washer.
- Vespel® ring embedded in top surface reduces operator variability by requiring minimal torque to seal.
- Prevents oxygen from entering the carrier gas, increasing column lifetime.

In Agilent split/splitless injection ports, our Dual Vespel® Ring Inlet Seal greatly improves performance, relative to conventional metal-to-metal seals—it stays sealed, even after repeated temperature cycles, without retightening the reducing nut! Two soft Vespel® rings, outside the sample flow path, eliminate the need for a washer and ensure very little torque is needed to make a leak-tight seal. Tests show Dual Vespel® Ring Inlet Seals seal equally effectively at torques from 5 in. lb. to 60 in. lb.

Use a stainless steel seal for analyses of unreactive compounds. To reduce breakdown and adsorption of active compounds, use a Siltek®-treated or gold-plated seal.•

| 0.8mm ID Dual Vespel® Ring Inlet Seal | 2-pk. | 10-pk. |
|---|---|---|
| Siltek® | 21242 | 21243 |
| Gold-Plated | 21240 | 21241 |
| Stainless Steel | 21238 | 21239 |
| **1.2mm ID Dual Vespel® Ring Inlet Seal** | **2-pk.** | **10-pk.** |
| Siltek® | 21248 | 21249 |
| Gold-Plated | 21246 | 21247 |
| Stainless Steel | 21244 | 21245 |

## Replacement Inlet Seals or Agilent 5890/6890/6850 Split/Splitless Injection Ports

- Special grade of stainless steel that is softer and deforms more easily, creating a better seal.
- Increases column lifetime because oxygen cannot permeate into the carrier gas.
- Reduced noise benefits high-sensitivity detectors (e.g., ECDs, MSDs).
- Siltek® treatment provides inertness similar to fused silica.
- All seals include washers.

The inlet seal at the base of the Agilent 5890/6890 GC injection port contacts the sample and, because septum fragments and sample residue accumulate on the seal surface, the seal must be changed frequently to prevent adsorption of active compounds.•

• Use a 1.2mm inlet seal with Vespel®/graphite ferrules or when installing two columns using a two-hole ferrule. Use a 0.8mm inlet seal with graphite ferrules or single capillary column installations.

| Single-Column Installation, 0.8mm Opening* | | 0.25/0.32mm ID Dual-Column Installation, 1.2mm Opening | | 0.53mm ID Dual-Column Installation (1/16-inch opening) | |
|---|---|---|---|---|---|
| 2-pk. | 10-pk. | 2-pk. | 10-pk. | 2-pk. | 10-pk. |
| | | **Stainless Steel Inlet Seal** | | | |
| 21315 | 21316 | 20390 | 20391 | 20392 | 20393 |
| | | **Gold-Plated Inlet Seal** | | | |
| 21317 | 21318 | 21305 | 21306 | — | — |
| | | **Siltek® Inlet Seal** | | | |
| 21319 | 21320 | 21307 | 21308 | — | — |

*0.8mm ID stainless steel inlet seal is similar to Agilent part #18740-20880, 0.8mm ID gold-plated inlet seal is similar to Agilent part #18740-20885.

## Replacement Inlet Seal Washers

| Description | Similar to Agilent part # | qty. | cat.# |
|---|---|---|---|
| Replacement Inlet Seal Washers | 5061-5869 | 15-pk. | 21710 |

# COOL TOOLS!

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## Capillary Installation Gauge

- Seats ferrule onto column for consistent installation.
- Prevents crushed column ends.
- Made from high-quality stainless steel.

For Agilent-style fittings

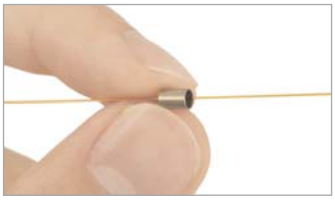
For standard 1/16" fittings

For TRACE™ 2000/8000 fittings

new for 2005

Install the nut and ferrule onto the column. Cut the column end squarely. Slide the column into the installation gauge to the recommended insertion distance. Finger-tighten the column nut.

Tighten the assembly to ensure a properly seated ferrule. Loosen the assembly and remove the column and column nut.



The ferrule will be properly seated, and should remain in place when light force is applied. If it slides loosely on the column, repeat procedure.

restek
**innovation**

Easily seat ferrules for consistent installations!

| Description | qty. | cat.# |
|---|---|---|
| Capillary Installation Gauge for Agilent-style fittings (compact ferrules)* | ea. | 21034 |
| Capillary Installation Gauge for 1/16" fittings (1/16" ferrules)* | ea. | 21399 |
| Capillary Installation Gauge for TRACE™ 2000/8000 (M4 Ferrules) | ea. | 22330 |

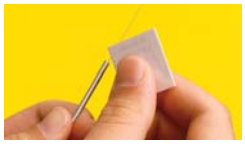
*Recommended for use with graphite ferrules.

## Capillary Installation Gauge for Agilent 5973 MS

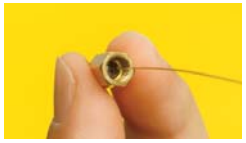
- Seats ferrules onto column for consistent installations.
- Made from high-quality stainless steel.

Install the nut and ferrule onto the column, then insert the column through the installation tool, exposing several centimeters at the exit end.

Tighten the nut.



Score and remove the exposed end of the column, then loosen the nut.



The ferrule will be properly seated and should remain in place when light force is applied. Install the column into the GC/MS interface.

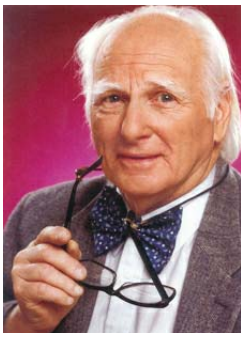
restek
**innovation**

Easily seat ferrules for consistent installations in Agilent 5973 MS.

| Description | Similar to Agilent part # | qty. | cat.# |
|---|---|---|---|
| Capillary Installation Gauge for Agilent 5973 MS | G1099-20039 | ea. | 21894 |

# Coming Soon...

## Articles by Walt Jennings Beginning in *The Restek Advantage*, 2006, Volume 1



Restek has a proud tradition of educating scientists about chromatography. Our seminars are highly valued by chemists around the world, and many practicing chromatographers look forward to receiving and reading *The Restek Advantage*.

We are very excited to announce that one of the pioneers in gas chromatography - Professor Walter Jennings - has offered to support our education efforts by periodically contributing articles to the *Advantage*.

Walt, as he is affectionately known around the globe, is a world-renowned authority in this field. He has authored many books, written countless research articles, and presented lectures to tens of thousands of chromatographers world-wide. Through many years of teaching at the University of California at Davis, he has developed a unique style of explaining complex technical issues in a way everyone can understand. Walt and Milton Lee share the 2005 California Separation Science Society (CaSSS) Award for Distinguished Contributions to Separation Science - the latest of many awards in two highly distinguished careers.

Walt is preparing an article for *The Restek Advantage*, 2006, volume 1, so you won't want to miss this next issue. Look for it in your mail box in February.

## **free** literature

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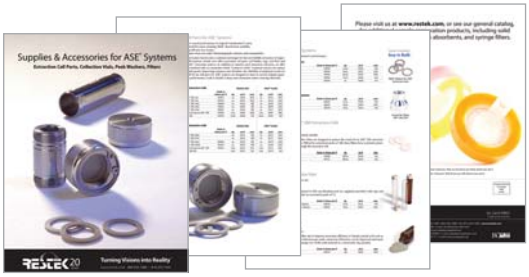


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